Quality Procedure Interim Change Notice

Date: **12/1/03** **Reviewed 10/3/05 (ER2005-0732)**

6 Page(s)

Section 1: Description of Change (Requester completes)		1. Document Catalog No.: **ER2003-0666**
2. QP & Rev. No.: **SOP-15.07, R1**	3. ICN No.: **1**	4. QP Title: **Routine Validation of Chemical Separation alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data**

5. Description of Change:

Section 6.2, step 2:

1) Delete 1st row in table and change < the MDC (1st colume) to ≤ the MDC.

2) Step 3: delete 1st row and change 2nd row from "<3 time" to "≤3 times."

3) Add the following text and table after step 3:
"Reference the following table as applicable."

MDC VALUE	SAMPLE VALUE	UNCERTAINTY VALUE	FU4 QUALIFIER REASON CODE
0	0	0	R, R5b
0	0	NULL	R, R5b
NULL	X-VALUE	0	R, R5a
NULL	0	0	R, R5a

4) Change Attachment B, page 32, 5th row, Reason Code R5, Description: "The results for affected analytes are considered not detected (U) because the associated sample concentration was less than or equal to the MDC."

5) Change Attachment B, page 33, 7th row, Rad R11: "The results of the affected analytes are considered not detected (U) because the associated sample concentration was less than or equal to three times the total propagated uncertainty."

6) Change Attachment D, number 2 as follows: "The sample result is less than or equal to the MDC."

7) Change Attachment D, number 3 as follows: "The sample result is less than or equal to three times the TPU calculation."

6. Attachments Modified, Added, or Removed: ☒ Yes ☐ No

7. ICN Justification:
Updating SOP to address MDA/sample value/uncertainty changes.

8. Requester: Nita Patel [Signature on file.] 11/20/03
(Print name, then sign.) (Date)

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Section 2: Evaluation and Approval (PTL, Technical Reviewer, and QPPL complete.)

9. Evaluation Remarks: (If none, enter N/A) N/A

10. Project Team Leader: Sheila Zhang [Signature on file.] 11/24/03
(Print name, then sign.) (Date)

11. Technical Reviewer: Keith Greene [Signature on file.] 11/20/03
(Print name, then sign.) (Date)

12. QPPL: Larry Maassen [Signature on file.] 11/25/03
(Print name, then sign.) (Date)

QP-4.1, R5	**Los Alamos National Laboratory** **RRES-Remediation Services Project**

Using a token card, click here to record "self-study" training to this procedure.

If you do not possess a token card or encounter problems, contact the RRES-ECR training specialist

Attachment B: Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data-Validation Reason Codes

Universal Reason Codes

Code	Rad	Qualifier Nondetects	Qualifier Detects	Description	Comments
1	R1	R	R	The required tracer/carrier information is missing. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
1a	R1a	R	R	The results for the affected analytes are qualified as rejected (R) because the associated tracer recovery was less than 10%.	
1b	R1b	N/A	J-	The results for the affected analytes are qualified as estimated and biased low (J-) because the associated tracer recovery was less than 30% but greater than 10%.	This code is used for detected analytes.
1c	R1c	UJ	N/A	The reporting limits for the affected analytes are qualified as estimated (UJ) because the associated tracer recovery was less than 30% but greater than 10%.	This code is used for undetected analytes.
1d	R1d	N/A	J+	The results for the affected analytes are qualified as estimated and biased high (J+) because the associated tracer recovery was greater than 105%.	Qualify only detected results.
3	R3	R	R	The required matrix spike information is missing.	The package should be returned to the SMO, or the information should be requested from the laboratory.
3a	R3a	N/A	R	The results for the affected analytes are considered rejected (R) because the associated matrix spike recovery was less than 10%.	This code is used for detected analytes.
3b	R3b	R	N/A	The reporting limits for the affected analytes are considered rejected (R) because the associated matrix spike recovery was less than 10%.	This code is used for undetected analytes.

Universal Reason Codes

Code	Rad	Qualifier Nondetects	Qualifier Detects	Description	Comments
3c	R3c	N/A	J+	The results for the affected analytes are considered estimated and biased high (J+) because the associated matrix spike recovery was above the UAL.	Qualify only detected results.
3d	R3d	N/A	J-	The results for the affected analytes are considered estimated and biased low (J-) because the associated matrix spike recovery was less than the lower acceptance limit (LAL) but greater than 10%.	This code is used for detected analytes.
3e	R3e	UJ	N/A	The reporting limits for the affected analytes are considered estimated (UJ) because the associated matrix spike recovery was less than the LAL but greater than 10%.	This code is used for (undetected) analytes.
4	R4	R (See comments)	R (See comments)	The required method-blank documentation is missing. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
4a	R4a	N/A	U	The results for the affected analytes are considered not detected (U) because the associated sample concentration was less than or equal to five times the amount in the method blank.	
5	R5	U	U	The results for the affected analytes are considered not detected (U) because the associated sample concentration was less than or equal to the MDC.	
5a	R5a	R (See comments)	R (See comments)	The MDC or TPU documentation is missing. Validation cannot proceed without this information.	The package should be returned to the SMO or the information requested from the laboratory.
6	R6	R (See comments)	R (See comments)	The LCS documentation is missing. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
6a	R6a	N/A	J+	The results for the affected analyte are considered estimated and biased high (J+) because the associated analyte in the LCS was recovered above the UAL.	Qualify only detected results.
6b	R6b	R	R	The results/reporting limits for the affected analytes should be regarded a rejected (R) because the associated LCS failed less than 10%.	

Universal Reason Codes

Code	Rad	Qualifier Nondetects	Qualifier Detects	Description	Comments
6c	R6c	N/A	J-	The results for the affected analyte are considered estimated and biased low (J-) because the associated LCS failed low but greater than 10%.	This code is used for detected analytes.
6d	R6d	UJ	N/A	The reporting limits for the affected analyte are considered estimated (UJ) because the associated LCS failed low but was greater than 10%.	This code is used for undetected analytes.
7	R7	UJ	J	The duplicate documentation is missing. Validation cannot determine the precision of the analysis without this information.	The package should be returned to SMO, or the information should be requested from the laboratory.
7a	R7a	N/A	J	The results for the affected analytes are qualified as estimated (J) because the associated duplicate sample was not prepared separately for the initial analysis.	
7b	R7b	N/A	J	The results for the affected analytes are qualified as estimated (J) because the associated duplicate sample has a DER of greater than two but less than four.	
7c	R7c	N/A	R	The results for the affected analytes are qualified as rejected (R) because the associated duplicate sample has a DER of greater than four.	
9	R9	UJ	J-	The results/reporting limits for the affected analytes are considered estimated and biased low (J-)/estimated (UJ) because the extraction holding time was exceeded.	
9a	R9a	R	R	The results for the affected analytes are considered rejected (R) because the sample extraction was double the method-published holding time.	
11	R11	N/A	U	The results for the affected analytes are considered not detected (U) because the associated sample concentration was less than or equal to three times the total propagated uncertainty.	This code should only be used after checking the detection status for results greater than the MDC.
19	R19	(See comments)	(See comments)	The validator has identified quality deficiencies in the reported data that require qualification. Please see the Data-Validation Cover Sheet for specific details.	Apply the appropriate qualifier to identify the effect of the quality deficiency on the reported data.

Attachment D: RAD Checklist

Yes	No	N/A	(check one)	Assign qualifier listed below if criteria = Yes	
				Detected analyte	Undetected analyte
☐	☐	☐	1. The MDC and/or TPU were not stated for each radionuclide in each batch for each sample.	(See note)[a]	(See note)[a]
☐	☐	☐	2. The sample result is less than or equal to the MDC.	U, R5	U, R5
☐	☐	☐	3. The sample result is less than or equal to three times the TPU calculation.	U, R11	N/A
☐	☐	☐	4. Required blank information is missing.	R, R4	R, R4
☐	☐	☐	5. The analyte detected is shown in the blank and sample result for analyte is less than or equal to the amount in blank.	U, R4a	N/A
☐	☐	☐	6. The duplicate analysis information is not present.	J, R7	UJ, R7
☐	☐	☐	7. The duplicate analysis was performed in lieu of a preparation duplicate.	J, R7a	N/A
☐	☐	☐	8. The DER for detected analytes is greater than but less than or equal to four.	J, R7b	N/A
☐	☐	☐	9. The DER for detected analytes is greater than four.	R, R7c	N/A
☐	☐	☐	10. The tracer/carrier information is not present.	R, R1	R, R1
☐	☐	☐	11. The tracer/carrier %R is greater than the UAL.	J+, R1d	N/A
☐	☐	☐	12. The tracer/carrier %R is less than 30 but greater than or equal to 10%.	J-, R1b	UJ, R1c
☐	☐	☐	13. The tracer/carrier %R is less than 10%.	R, R1a	R, R1a
☐	☐	☐	14. The LCS information is not present.	R, R6	R, R6
☐	☐	☐	15. The LCS %R is greater than 120%.	J+, R6a	N/A
☐	☐	☐	16. The LCS %R is less than 80 but greater than or equal to 10%.	J-, R6c	UJ, R6d
☐	☐	☐	17. The LCS %R is less than 10%.	R, R6b	R, R6b
☐	☐	☐	18. The matrix spike information is not present.	R, R3	R, R3
☐	☐	☐	19. The matrix spike %R is greater than 125%.	J+, R3c	N/A
☐	☐	☐	20. The matrix spike %R is less than 75% but greater than or equal to 10%.	J-, R3d	UJ, R3e
☐	☐	☐	21. The matrix spike %R is less than 10%.	R, R3a	R, R3b
☐	☐	☐	22. The sample was extracted after the appropriate holding time was exceeded.	J-, R9	UJ, R9
☐	☐	☐	23. The sample was extracted after a period double the appropriate holding time was exceeded.	R, R9a	R, R9a
☐	☐	☐	24. Other obvious data quality issues are identified.	__, R19	__, R19

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Author: **Kim Hejde**	

RRES Remediation Program

Risk Reduction and Environmental Stewardship—Remediation Program

Standard Operating Procedure

for Routine Validation of Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data

Los Alamos NATIONAL LABORATORY
Los Alamos NM 87545

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Revision Log

Revision No.	***Effective Date***	***Prepared By***	***Description of Changes***	***Affected Pages***
R0	07/20/00	Bart Vanden Plas	Initial Procedure	All
R1	05/19/03	Kim Hejde	Rewritten to streamline and update process.	All
ICN1	12/1/03	N. Patel	Change to attachments	12-14,31-33,35
Reviewed	3/30/04	N. Patel`	Deemed adequate	
Reviewed	10/3/05	N. Patel	Deemed adequate	

Table Of Contents

Routine Validation of Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data

1.0 PURPOSE

1.1 This standard operating procedure (SOP) represents the minimum standards for evaluating routine radionuclide analytical data generated for the Los Alamos National Laboratory (LANL), Risk reduction and Environmental Stewardship—Remediation Program (RRES-R) for samples analyzed for,

- Alpha-emitting isotopes (americium-241; uranium-234, -235, and -238; thorium -230, -232 and -234 and plutonium-238 and -239/-240) by chemical separation alpha spectrometry,
- Strontium-90 by gas proportional counting (GPC),
- Gross alpha and beta analyses by GPC, and
- Tritium by liquid scintillation.

1.2 These evaluations must follow the methods required under the current statement of work (SOW) for analytical services (LANL, 1995). Because the various technologies of each method carry specific requirements, the validation procedure in Section 6.0 explains how to apply relevant requirements to the methods and isotopes explained in each subsection.Data evaluation in this procedure is not specific to a particular data use, although this procedure may be used to develop focused data-validation requirements specific to a particular data use.

1.3 This procedure tabulates data compliances and noncompliances identified relative to expectations based on national guidelines (U.S. Environmental Protection Agency [EPA], 1994a; 1994b) for data review. Data noncompliance is indicated by applying qualifiers (Attachment A) and reason codes (Attachment B) to reported results. Because EPA guidelines are specific to inorganic/organic chemical analyses, additional guidance (ANSI 1996; Currie 1968; Fong and Alvarez 1996; MARSSIM, 1997; and LANL, 1995) was used to prepare this SOP. Because the acceptance criteria used for this procedure are not based on site-specific acceptance criteria, the validation procedure results are intended to indicate data quality and not data usability.

1.4 Nothing in this SOP precludes the validator from going beyond the minimum requirements specified in this SOP. In order to address data quality issues in a data package, the validator may assign qualifiers using

professional judgment. This procedure may be followed by a more focused and data-use-specific evaluation of data, especially if results of this SOP indicate that technical deficiencies may exist in the data. The validator will note any need for a more focused validation on the Data-Validation Cover Sheet (Attachment C). The validator will use the Data-Validation Checklist (Attachment D) to record the specific validation steps conducted.

2.0 SCOPE

2.1 All **RRES-R Personnel** shall implement this mandatory SOP when evaluating routine radionuclide analytical data.

2.2 Subcontractors performing work under the RRES-R Program's quality program shall follow this SOP.

3.0 TRAINING

3.1 **RRES-R Personnel** shall train to and use the current version of this SOP; contact the author if the SOP text is unclear.

3.2 **RRES-R Personnel** using this SOP shall document training in the RRES-R training database located at **http://erinternal.lanl.gov/Training/login.asp** in accordance with QP-2.2.

3.3 The responsible **supervisor** shall monitor the proper implementation of this procedure and ensure that the appropriate personnel complete all applicable training assignments.

3.4 **Data validators** who implement this SOP shall

- possess a minimum of a bachelor's degree in chemistry or one of the physical sciences and two years of experience in generating analytical data in an environmental analytical laboratory, or two years of data-validation experience;
- if inexperienced, work under the direct supervision of an experienced RRES-R validator, who reviews and signs off on work until 10 data packages demonstrate satisfactory validation for each analytical suite; and
- demonstrat familiarity with the EPA national functional guidelines for data review (EPA 1994a; 1994b).

4.0 DEFINITIONS

4.1 *Activity concentration*—Level of radioactivity per unit volume or mass measured as a concentration; usually reported in pCi/g or pCi/L.

4.2 *Analyte*—The element, nuclide, or ion that a chemical analysis seeks to identify and/or quantify; the chemical constituent of interest.

4.3 *Blank sample*—Sample expected to have negligible or unmeasurable amounts of analytes. Results of blank-sample analyses indicate whether field samples might have been contaminated during the sample collection, transport, storage, preparation, and analysis process.

4.4 *Data validator*—A person who has met the minimum training standards established by the RRES-R Program for data validation and who performs data validation on behalf of the RRES-R Program (hereinafter referred to as the "validator").

4.5 *Detect (radionuclides)*—Sample result greater than the minimum detectable concentration (MDC) reported by the analytical laboratory. The laboratory reports the concentration of the analyte in the sample.

4.6 *Detector background*—Ambient signal response, recorded by radioactivity measuring instruments, that is independent of radioactivity contributed by the radionuclides being measured in the sample.

4.7 *Duplicate analysis*—Analysis performed on one of a pair of identically prepared subsamples taken from the same sample. The subsamples can be created in the field (field duplicate samples) or in the laboratory (laboratory duplicate samples).

4.8 *Duplicate error ratio (DER)*—Measure of precision of analytical laboratory duplicate samples in a batch. The DER is based on the standard deviations of the sample and the duplicate sample.

$$DER = \frac{|S - R|}{\sqrt{u_S^2 + u_R^2}} ,$$

where

DER = duplicate error ratio,

S = sample value,

R = duplicate value,

u_S = sample uncertainty, and

u_R = duplicate uncertainty.

Note: If DER is less than two, then the sample and duplicate are not statistically different at the 95% confidence level.

Note: The DER value is based on 1σ (standard deviation of a set of measurements). The validator should pay particular attention to the reporting practices of the laboratory and adjust the DER value accordingly (for example, if the laboratory reports the uncertainties as

2σ, the DER value should be recalculated using 1σ). The DER should also be calculated on a true duplicate not a reanalysis of the same sample.

4.9 *Form 1*—Organic Analysis Data Sheet for each individual sample that includes the sample information needed to identify the sample and the sample's analytical results. See the SOW for analytical services (RFP No. 9-XS1-Q4257) for a more complete definition.

4.10 *Holding time*—The maximum length of time that one can expect to store a sample without unacceptable changes in analyte concentrations. Holding times apply under prescribed conditions, and deviations from these conditions may affect the holding time. Extraction holding time refers to the time lapse from sample collection to sample preparation; analytical holding time refers to the time lapse between sample preparation and analysis.

4.11 *Laboratory control sample (LCS)*—A known matrix that has been spiked with compound(s) representative of the target analytes. The LCS is used to document laboratory performance. The acceptance criteria for LCSs are method-specific.

4.12 *Laboratory duplicate sample*—The portions of a sample taken from the same sample container, prepared for analysis and analyzed independently but under identical conditions; used to assess or demonstrate acceptable laboratory method precision during analysis. Each duplicate sample is expected to equally represent the original material. Duplicate analyses also are performed to generate data to determine the long-term precision of an analytical method on various matrices.

4.13 *LANL data-validation qualifiers*—The data qualifiers defined by LANL and used in the RRES-R Program routine validation process. Attachment A lists the data qualifiers applicable to all analytical suites.

4.14 *LANL data-validation reason codes*—The codes applied to the sample data by data validators who are independent of the contract laboratory that performed the sample analysis. Reason codes provide an in-depth and analysis-specific explanation for applying the qualifier, along with a description of the potential impact on the data use. For a complete list of data qualifiers applicable to any particular analytical suite, consult the appropriate RRES-R Program SOP.

4.15 *Matrix spike*—An aliquot of sample spiked with a known concentration of target analyte(s). Matrix-spike samples are used to measure the ability to recover prescribed analytes from a native sample matrix. The spiking typically occurs before sample preparation and analysis.

4.16 *Minimum detectable concentration* (MDC)—Minimum activity concentration that the analytical laboratory equipment can detect in 95% of the analyzed samples. That is, if the actual concentration of a sample is above MDC, a less than 5% chance exists that the measured concentration will fall below the DLC and result in a "nondetect." An MDC measures analytical performance (not detection limits),

$$MDC = C \times \left(2.71 + 4.65\sqrt{N_b}\right),$$

where

MDC = the minimum detectable concentration reported in pCi/g or pCi/L;

C = a group of factors that convert counts to an activity concentration (C is omitted if N_b is expressed in concentration units);

2.71 = 1.65^2 (1.65 normal probability, one sided, for 0.05 significance);

4.65 = $1.65 \times 2(2)^{0.5}$;

N_b = total analyte-free blank (or background) counts, and all blank, background, and sample count times are equal.

4.17 *Nondetect (radionuclides)*—A sample result that is less than the MDC.

4.18 *Percent recovery (%R)*—Amount of material detected in a sample (minus any amount already in the sample) divided by the amount added to the sample and expressed as a percentage.

4.19 *Precision*—Concept used to describe the dispersion of measurements. The precision may be absolute or relative to a particular measure of central tendency. The mathematical formulas used to determine precision vary according to the problem at hand.

4.20 *Preparation blank*—An analyte-free matrix to which all reagents are added in the same volumes or proportions as those used in the environmental sample processing. The preparation blank is prepared and analyzed in the same manner as the corresponding environmental samples and used to assess the potential for contamination of samples during preparation and analysis.

4.21 *Request number (RN)*—An identifying number assigned by the RRES-R Program to a group of samples submitted for analysis.

4.22 *Routine data*—Data generated using analytical methods that are identified as routine methods in the current RRES-R Program SOW for analytical services.

4.23 *Routine radionuclide data*—Analytical results and associated data for samples analyzed for alpha-emitting isotopes (by chemical-separation

alpha spectrometry), strontium-90 (gas proportional counting [GPC]), gross alpha and beta analyses (GPC), and tritium (liquid scintillation). Routine validation of gamma spectroscopy is included in SOP-15.06 but not in SOP-15.07 because of the greater complexity of gamma spectroscopy data.

4.24 *Routine data validation*—Reviewing analytical data relative to quantitative routine acceptance criteria. The objective of routine data validation is twofold: one objective is to estimate the technical quality of the data relative to minimum national guidelines adopted by the RRES-R Program; the other objective is to show data users the technical data quality at a general level by assigning qualifier flags to environmental data whose quality indicators do not meet acceptance criteria.

4.25 *Sigma (σ)*—Standard deviation (square root of the variance) of a set of measurements. For normally distributed data, a range of one sigma (1σ) below the estimated mean to one sigma (1σ) above the estimated mean signifies a 67% confidence that the mean of a population lies within that range. Similarly, a range of plus/minus two sigma ($\pm 2\sigma$) implies a 95% confidence that a population mean lies within that range.

4.26 *Target analyte*—An element, chemical, or parameter of which the concentration, mass, or magnitude is designed to be quantified with a particular test method.

4.27 *Total propagated uncertainty (TPU)*—Sum of all aspects of uncertainty introduced throughout the sample analysis process, from sample collection to reporting of results. Many aspects of TPU may be specifically calculated by an analytical laboratory (e.g., net instrumental error, counting uncertainty). Other aspects of TPU may not be quantifiable (e.g., heterogeneity of concentrations at site), and thus cannot be directly included in a laboratory's estimate of TPU.

5.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure:

- Data Validator (see definition 4.4.)
- RRES-R Personnel
- Quality Program Project Leader
- Supervisor
- User

6.0 PROCEDURE

Make any deviations from this SOP in accordance with QP-5.7 and/or SOP-01.01. The **data validator** perform all steps in this procedure unless otherwise indicated.

Note: Although this SOP is applicable to chemical-separation alpha spectrometry (for alpha-emitting isotopes), GPC (for strontium-90 and gross α/β), and liquid scintillation (for tritium), each subsection does not apply equally to each method. Notes at the beginning of each subsection describe how each subsection applies to each method.

6.1 Preparing for Data Validation

Section 6.1 applies to all routine radionuclide analysis methods and analytes covered by this SOP.

1. Obtain the required current version of the Routine Radionuclide Data-Validation Checklist form (Attachment D) from the RRES-R Program website (http://erinternal.lanl.gov/Quality/forms.htm).

   A. Obtain from the Sample Management Office (SMO) of the Field Support Facility (FSF) the data-record package(s) that contains the sample data to be validated.

   B. Prepare a Data-Validation Cover Sheet (Attachment C) by completing the top part of the form and placing a check or other mark adjacent to the analytical suites for which this validation is being performed.

   C. If data is rejected, the rejected box will be checked and the project chemist will be notified immediately.

      Note: A single cover sheet may be used for validation of multiple analytical suites under the same request number (RN).

      Note: Use a separate sheet of paper to document each deficiency identified beyond the scope of this procedure, including telephone conversations with the analytical laboratory personnel concerning these deficiencies. Attach these sheets to the Data-Validation Cover Sheet.

2. Verify that the following items are present in the data-record package:

   - Signed LANL chain of custody (COC) record
   - Case narrative
   - Result forms (Contract Laboratory Program [CLP] Form 1 or equivalent) for each sample

- Quality control (QC) forms (CLP forms, or equivalent) for water and/or soils, as appropriate
- Instrument readout (raw data) for the samples

3.

IF the required documentation for the data-record package is...	**FOR...**	**THEN...**
Complete,		• Go to Step 5.
Missing,	< 6 mo.	• Contact the analytical laboratory and/or the SMO. • Allow 3 business days for submittal. • Go to Step 4.
Missing,	≥ 6 mo.	• Contact the analytical laboratory and/or the SMO. • Allow 10 business days for submittal. • Go to Step 4.

Note: To expedite the validation process, the validator may request that the contract laboratory forward the missing information directly to the validator by fax or e-mail within 24 h of notification.

4.

IF the analytical laboratory...	**THEN...**
Submits the documentation within the specified period of time,	• Go to Step 5.
Does <u>not</u> submit the documentation within the specified period of time,	• Notify the SMO for contract-compliance action. • Go to Step 5.

5. Record the presence or absence (by checking “Yes” or “No”) of each item, as appropriate, in the completeness checklist of the Data-Validation Cover Sheet.

6. In the Data-Validation Cover Sheet completeness checklist section, note any samples whose data are missing from the data-record package.

7. Photocopy the following:

   - Form 1, the results report from the analytical laboratory that will be used during the validation process.
   - Chain of custody forms

     Note: Do not record data-validation qualifiers and reason codes on the original form (Form 1).

     Note: The validator must submit photocopies of all analytical laboratory QC forms, the case narrative, and Form 1 as attachments to the completed data-validation checklists. The validator must initial and date each page of Form 1; these initials and date must be present even if the validator accepts laboratory qualification.

6.2 Verifying Sample Detect Status and Validating Sample Results

Note: Whenever the required information is missing, the validator must notify the analytical laboratory and/or the SMO to obtain the information before validation can proceed.

Note: Section 6.2 applies to all routine radionuclide analysis methods and analytes. This analysis must be performed before subsequent procedure steps in order to determine the detection status for the target analytes.

1.

IF…	**THEN…**
The minimum detectable concentration (MDC) was stated in the report for each nuclide of each batch sample associated with this RN,	• Record “No” on line 1 of the Routine Radionuclide Data-Validation Checklist. • Go to Step 2.

1.

IF…	THEN…
Any MDC was not stated in the report for each nuclide of each batch sample associated with this RN,	• Notify the SMO and laboratory to request the missing information (see Section 6.1-4). • If the laboratory is unable to provide the missing information, record “Yes” on line 1 of the Routine Radionuclide Data-Validation Checklist. • Record the estimated (calculated) MDC for the affected analytes on the individual Form 1 using three times the 1 standard deviation (σ) total propagated uncertainty (TPU) of the sample result. • Go to Step 3.

2.

IF the sample value is…	THEN…
$>$ the MDC,	• Record "No” on line 2 of the Routine Radionuclide Data-Validation Checklist. • Go to Step 3.
$<$ the MDC,	• Record "Yes” on line 2 and “n/a” (not applicable) on line 3 of the Routine Radionuclide Data-Validation Checklist. • Qualify affected analytes as undetected (U, R5) on the individual sample Form 1s. • Go to Section 6.3, “Verifying the Blank Method Results.”

3.

IF the sample value is…	THEN…
≥ 3 times the 1σ TPU,	• Record "No" on line 3 of the Routine Radionuclide Data-Validation Checklist. • Go to Section 6.3, "Verifying Blank Method Results."
<3 times the 1σ TPU,	• Record "Yes" on line 3 of the Routine Radionuclide Data-Validation Checklist. • Qualify affected analytes as undetected (U, R11) on the individual sample Form 1s. • Go to Section 6.3, "Verifying Blank Method Results."

6.3 Verifying the Blank Method Results

Note: Verify the presence of the required blanks (preparation and/or method blanks) and associated results using forms provided by the analytical laboratory. The blank results should be less than the MDC for each nuclide.

Note: Section 6.3 applies to all routine radionuclide analysis methods and analytes covered by this SOP.

Note: If additional validation forms are needed to record validation data for more than one blank, make additional copies of the appropriate forms.

1.

IF…	THEN…
All required blank information is reported in the data package,	• Record "No" on line 4 of the Routine Radionuclide Data-Validation Checklist. • Go to Step 2.

1.

IF…	THEN…
Any required blank information is missing,	• Record "Yes" on line 4 of the Routine Radionuclide Data-Validation Checklist. • Contact the analytical laboratory and the SMO to obtain the missing information (see Section 6.1-4). • If the missing information is not provided, qualify all affected results as rejected (R, R4) on the individual sample Form 1s. • Go to Step 2.

2.

IF…	THEN…
All required blanks have no contamination or the results for all analytes in all samples are >5 times the concentration in the corresponding blank,	• Record "No" on line 5 of the Routine Radionuclide Data-Validation Checklist. • Go to Section 6.4, "Verifying the Laboratory Duplicate Results."
The concentration of any analyte in a sample is ≤ to 5 times the concentration of that analyte in the corresponding blank,	• Record "Yes" on line 5 of the Routine Radionuclide Data-Validation Checklist. • Qualify all affected analytes as undetected (U, R4a) on the individual sample Form 1s. • Go to Section 6.4, "Verifying the Laboratory Duplicate Results."

6.4 Verifying the Laboratory Duplicate Results

Note: Section 6.4 applies to all routine radionuclide analysis methods and analytes with the exception of ^{235}U. ^{235}U is not held to the DER limits because ^{235}U is typically detected at much lower levels than ^{234}U and ^{238}U.

Note: Verify the presence of the laboratory duplicate using the forms provided by the analytical laboratory. Verify that the duplicate analysis was

performed on a second preparation of the sample and not just a reanalysis of the original sample.

Note: Find the reported DER in the data-record package for the duplicate and the sample result, or calculate the DER using the equation in Section 4.8.

Note: If the DER is less than two, then the sample and replicate are not statistically different at the 95% confidence level.

Note: The DER limit is based on 1σ. The validator should pay particular attention to the reporting practices of the laboratory and calculate the DER accordingly (i.e., if the laboratory reports the results as 2σ, then recalculate the DER using 1σ).

1.

IF…	**THEN…**
All duplicate information is present in the data package,	• Record "No" on line 6 of the Routine Radionuclide Data-Validation Checklist. • Go to Step 2.
Any duplicate information is missing,	• Record "Yes" on line 6 of the Routine Radionuclide Data-Validation Checklist. • Contact the analytical laboratory and/or SMO to request the missing information (see Section 6.1-4). • Qualify all affected analytes as estimated (J, R7/UJ, R7) on the individual sample Form 1s. **Note:** If this data is missing, validation can proceed, but check with the SMO for contract compliance purposes. • Go to Step 2.

2.

IF…	**THEN…**
A duplicate was performed, the DER is <2 for all analytes and was performed on a second preparation of the sample,	• Record "No" on lines 7, 8, and 9 of the Routine Radionuclide Data-Validation Checklist. • Go to Section 6.5, "Verifying the

2.

IF…	THEN…
	Tracer/Carrier Recoveries."
There was an insufficient amount of sample to perform a preparation duplicate on a sample in the RN, and a duplicate analysis was performed on the sample in lieu of a preparation duplicate,	• Record "Yes" on line 7 of the Routine Radionuclide Data-Validation Checklist. • Qualify all detected analytes in affected samples as estimated (J, R7) on the individual sample Form 1s. • Go to Step 3.

3.

IF the duplicate and sample results for detected analytes have a DER that is…	THEN…
<2,	• Record "No" on lines 8 and 9 of the Routine Radionuclide Data-Validation Checklist. • Go to Section 6.5, "Verifying the Tracer/Carrier Recovery."
≥2 but ≤4,	• Record "Yes" on line 8 of the Routine Radionuclide Data-Validation Checklist. • Qualify all detected analytes in affected samples as estimated (J, R7b) on the individual sample Form 1s. • Go to Step 4.

4. For detected analytes, if the duplicate and sample results have a DER that is >4,
   A. Record "Yes" on line 9 of the Routine Radionuclide Data-Validation Checklist.
   B. Qualify all detected analytes in affected samples as rejected (R, R7c) on the individual sample Form 1s.
   C. Go to Section 6.5, "Verifying the Tracer/Carrier Recovery."

6.5 Verifying the Tracer/Carrier Recovery

Note: Section 6.5 only applies to chemical separation alpha spectrometry and GPC for strontium-90. This section does not apply to tritium by liquid scintillation or to gross α/β by GPC. For tritium and gross α/β, go to Section 6.6, "Verifying the Laboratory Control Sample Results."

1.

IF…	**THEN…**
<u>All</u> tracer/carrier information is <u>present</u> in the data package,	• Record "No" on line 10 of the Routine Radionuclide Data-Validation Checklist. • Go to Step 2.
Tracer/carrier information is <u>missing</u>,	• Record "Yes" on line 10 of the Routine Radionuclide Data-Validation Checklist. • Contact the analytical laboratory and/or the SMO to request the missing information (see Section 6.1-4). • If the tracer/carrier information is not provided, qualify all affected results as rejected (R, R1) on the individual sample Form 1s. • Go to 6.6, "Verifying the Laboratory Control Sample Results."

2.

IF…	**THEN…**
<u>All</u> tracer/carrier percent recoveries (%R) meet the acceptance criteria,	• Record "No" on lines 11, 12, and 13 of the Routine Radionuclide Data-Validation Checklist. • Go to Section 6.6, "Verifying the Laboratory Control Sample Results."
<u>No</u> tracer/carrier %R in a sample is > the upper acceptable limit (UAL),	• Record "No" on line 11 of the Routine Radionuclide Data-Validation Checklist. • Go to Step 3.

2.

IF…	THEN…
Any tracer/carrier %R in a sample is >UAL,	• Record "Yes" on line 11 of the Routine Radionuclide Data-Validation Checklist. • Qualify all detected analytes as estimated with a potential postive bias (J+, R1a) on the individual sample Form 1s. • Go to Step 3.

3.

IF…	THEN…
No tracer/carrier %R in a sample is <30%,	• Record “No” on lines 12 and 13 of the Routine Radionuclide Data-Validation Checklist. • Go to Section 6.6, “Verifying the Laboratory Control Sample.”
Any tracer/carrier %R in a sample is <30% but ≥ to 10%,	• Record “Yes” on line 12 of the Routine Radionuclide Data-Validation Checklist. • Qualify all detected analytes in affected sample as estimated with a potential negative bias (J-, R1b) and all undetected analytes in affected samples as estimated (UJ, R1c) on the individual sample Form 1s. • Go to Step 4.

4.

IF…	THEN…
No tracer/carrier %R in a sample is <10%,	• Record "No" on line 13 of the Routine Radionuclide Data-Validation Checklist. • Go to Section 6.6, "Verifying the Laboratory Control Sample Results."
Any tracer/carrier %R in a sample is <10%,	• Record "Yes" on line 13 of the Routine Radionuclide Data-Validation Checklist. • Qualify all affected analytes as rejected (R, R1a) on the individual sample Form 1s. • Go to Section 6.6, "Verifying the Laboratory Control Sample Results."

6.6 Verifying the Laboratory Control Sample Results

Note: Section 6.6 applies to all routine radionuclide analysis methods and analytes. Verify the presence of the laboratory control sample (LCS) %R values using forms provided by the analytical laboratory. The LCS acceptance criteria are 80%–120%, inclusive, for all spiked analytes.

1.

IF…	THEN…
All LCS information is provided in the data package,	• Record "No" on line 14 of the Routine Radionuclide Data-Validation Checklist. • Go to Step 2.
Any LCS information is missing,	• Record "Yes" on line 14 of the Routine Radionuclide Data-Validation Checklist. • Contact the analytical laboratory and/or the SMO for contract compliance action (see Section 6.1-4). • If the LCS information is not provided, qualify all affected analytes as rejected (R, R6) on the individual sample Form 1s.

1.

IF…	THEN…
	• Go to Step 2.

2.

IF an LCS %R value …	THEN…
Meets the acceptance criteria,	• Record "No" on lines 15, 16, and 17 of the Routine Radionuclide Data-Validation Checklist. • Go to Section 6.7, "Verifying the Matrix Spike Results."
Does <u>not</u> meet the acceptance criteria,	• Go to Step 2.

3.

IF…	THEN…
<u>No</u> LCS %R in a sample is >120%,	• Record "No" on line 15 of the Routine Radionuclide Data-Validation Checklist. • Go to Step 4.
<u>Any</u> LCS %R is >120%,	• Record "Yes" on line 15 of the Routine Radionuclide Data-Validation Checklist. • Qualify all detected analytes as estimated with a potential positive bias (J+, R6a) on the individual sample Form 1s. • Go to Step 4.

4.

IF…	THEN…
<u>No</u> LCS %R in a sample is <80%,	• Record "No" on lines 16 and 17 of the Routine Radionuclide Data-Validation Checklist. • Go to Section 6.7, "Verifying the Matrix Spike Results."

4.

IF…	THEN…
The LCS %R is <80% but ≥10%,	• Record "Yes" on line 16 of the Routine Radionuclide Data-Validation Checklist. • Qualify all detected analytes as estimated with a potential negative bias (J-, R6c) and all undetected analytes as estimated (UJ, R6d) on the individual sample Form 1s. • Go to Step 5.

5.

IF…	THEN…
No LCS %R is <10%,	• Record "No" on line 17 of the Routine Radionuclide Data-Validation Checklist. • Go to Section 6.7, "Verifying the Matrix Spike Results."
Any LCS %R is <10%,	• Record "Yes" on line 17 of the Routine Radionuclide Data-Validation Checklist. • Qualify all detected analytes as rejected (R, R6b) on the individual sample Form 1s. • Go to Section 6.7, "Verifying the Matrix Spike Results."

6.7 Verifying the Matrix Spike Results

Note: Section 6.7 applies only to test methods in which enough aliquot was provided for the matrix spike analyses. If the matrix spike was not performed for allowable reasons (not required by the requestor or insufficient sample provided) record "n/a" on lines 18, 19, 20, and 21 of the Routine Radionuclide Data-Validation Checklist and go to Section 6.8, Verifying Holding Time. Record why the MS/MSD was not required on the Data-Validation Cover Sheet. The matrix-spike acceptance criteria are 75%–125%, inclusive, for all spiked analytes. If the sample result is more than 4 times the amount of the spike added, these acceptance criteria do not apply.

1.

IF the MS/MSD information is…	**THEN…**
Provided in the data package as required,	• Record "No" on line 18 of the Routine Radionuclide Data-Validation Checklist. • Go to Step 2.
Missing,	• Record "Yes" on line 18 of the Routine Radionuclide Data-Validation Checklist. • Contact the analytical laboratory and/or the SMO to request the missing information (see Section 6.1-4). • If the MS/MSD information is not provided, qualify all affected results as rejected (R, R3) on the individual sample Form 1s. **Note:** If this data is missing, validation can proceed, but check with the laboratory for consistency purposes. • Go to Step 2.

2.

IF all MS/MSD %R …	**THEN…**
Meet the acceptance criteria,	• Record "No" on lines 19, 20, and 21 of the Routine Radionuclide Data-Validation Checklist. • Go to Section 6.8, "Verifying the Holding Time."
Do <u>not</u> meet the acceptance criteria,	• Go to Step 3.

3.

IF…	THEN…
No MS/MSD %R in a sample is >125%,	• Record "No" on line 19 of the Routine Radionuclide Data-Validation Checklist. • Go to Step 4.
Any MS/MSD %R is >125%,	• Record "Yes" on line 19 of the Routine Radionuclide Data-Validation Checklist. • Qualify all detected analytes as estimated with a potential positive bias (J+, R3c) on the individual sample Form 1s. • Go to Step 4.

4.

IF…	THEN…
No MS/MSD %R in a sample is <75%,	• Record "No" on lines 20 and 21 of the Routine Radionuclide Data-Validation Checklist. • Go to Section 6.8, "Verifying the Holding Time."
Any MS/MSD %R is <75% but ≥10%,	• Record "Yes" on line 20 of the Routine Radionuclide Data-Validation Checklist. • Qualify all detected analytes as estimated with a potential negative bias (J-, R3d) and all undetected analytes as estimated (UJ, R3e) on the individual sample Form 1s. • Go to Step 5.

5.

IF…	THEN…
No MS/MSD %R is <10%,	• Record "No" on line 21 of the Routine Radionuclide Data-Validation Checklist. • Go to Section 6.8, "Verifying the Holding Time."

5.

IF…	THEN…
Any MS/MSD %R is <10%,	• Record "Yes" on line 21 of the Routine Radionuclide Data-Validation Checklist. • Qualify all affected analytes as rejected (R, R3a for detected analytes and R, R3b for undetected analytes) on the individual sample Form 1s. • Go to Section 6.8, "Verifying the Holding Time."

6.8 Verifying the Holding Time

1.

IF…	THEN…
All samples were analyzed within their holding times,	• Record "No" on lines 22 and 23 of the Routine Radionuclide Data-Validation Checklist. • Go to Section 6.9, "Identifying the Obvious Quality Deficiencies."
Any samples were analyzed beyond the holding time,	• Record "Yes" on line 22 of the Routine Radionuclide Data-Validation Checklist. • Calculate the number of days the holding time was exceeded. • Go to Step 2.

2.

IF the holding time was more than…	THEN…
The required holding time, but <2 times the required holding time,	• Record "No" on line 23 of the Routine Radionuclide Data-Validation Checklist. • Qualify all detected analytes as estimated with a potential negative bias (J-, R9) and all undetected analytes as estimated (UJ, R9) on the individual sample Form 1s.

2.

IF the holding time was more than…	THEN…
	• Go to Section 6.9, “Identifying the Obvious Quality Deficiencies.”
Two times the required holding time,	• Record "Yes” on line 23 on the Routine Radionuclide Data-Validation Checklist. • Qualify all affected analytes as rejected (R, R9a) on the individual sample Form 1s. • Go to Section 6.9, “Identifying the Obvious Quality Deficiencies.”

6.9 Identifying the Obvious Quality Deficiencies

IF…	THEN…
Any significant or obvious data quality deficiencies are noticed during the data-validation process,	• Record “Yes” on line 24 of the Routine Radionuclide Data-Validation Checklist. • Contact the analytical laboratory and SMO, if necessary, to resolve the quality issue. • Apply the appropriate qualifier to the data based on the validator’s best professional judgment and apply reason code R19. • Write up a clear description of the quality issue on the Data-Validation Cover Sheet. • Go to Section 6.10, “Assembling the Data-Record Package.”
There are no obvious quality deficiencies outside of those covered by this SOP,	• Record “No” on line 24 of the Routine Radionuclide Data-Validation Checklist. • Go to Section 6.10, “Assembling the Data-Record Package.”

6.10 Assembling and Submitting the Data-Record Package

1. Assemble the validation data-record package to include the following items in the order listed below:
   - The completed, signed, and dated Data-Validation Cover Sheet
   - The Routine Radionuclide Data-Validation Checklist forms completed in Sections 6.2 through 6.6
   - Photocopies of the completed forms (Form 1) on which the data validator recorded the qualifier flags and reason codes
   - A photocopy of the data-record package case narrative
   - Photocopies of the data-record package QC forms (assembled in order by QC form)
2. Submit the validation data-record package to the SMO, in accordance with SOP-15.09, Chain of Custody for Analytical Data Packages.

7.0 LESSONS LEARNED

7.1 Before performing work described in this SOP, **RRES-R Personnel** should go to the Department of Energy Lessons Learned Information Services home page, located at http://www.tis.eh.doe.gov/ll/ll.html, and/or to the LANL Lessons Learned Resources web page, located at http://www.lanl.gov/projects/lessons_learned/, and search for applicable lessons.

7.2 During work performance and/or after the completion of work activities, **RRES-R Personnel**, as appropriate, shall identify, document, and submit lessons learned in accordance with the LANL, Lessons Learned System located at http://www.lanl.gov/projects/lessons_learned/.

8.0 RECORDS

Although no records are submitted to the Records Processing Facility (RPF) after this procedure is completed, the items identified in Section 8.8 are included in the data-record package submitted to the RPF from the SMO, in accordance with SOP-15.09.

9.0 REFERENCES

To properly implement this SOP, **RRES-R Personnel** should become familiar with the contents of the following documents located at http://erinternal.lanl.gov/home_links/Library_proc.shtml:

- “Measurement and Associated Instrumentation Quality Assurance for Radioassay Laboratories,” ANSI N42.23-1996 (American National Standards Institute, Inc., New York, 1996).
- L. Currie, “Limits for Qualitative Detection and Quantitative Determination, Application to Radiochemistry,” *Anal. Chem.* **40**, No. 3 (March 1968).
- RRES-R Program, RRES-R Program Quality Management Plan, ER Project QP Requirements Crosswalk, http://erinternal.lanl.gov/documents/Procedures/qps.htm.
- “U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review,” Publication 9240.1-05-01, EPA-540/R-94/013 (U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C., 1994)
- “U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review,” Publication 9240.1-05, EPA-540/R-94/012 (U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C., 1994).
- "Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM)," EPA 402-R-97-016, NUREG-1575 (U.S. Environmental Protection Agency, Washington, D.C., 1997)
- S. Fong and J. Alvarez, “Data Quality Objectives for Surface-Soil Cleanup Operation Using *In Situ* Gamma Spectrometry for Concentration Measurements,” *Health Physics* **72**, No. 2 (February 1997).
- “RRES-R Program Statement of Work for Analytical Services,” Revision 2, Los Alamos National Laboratory RFP Number 9-SX1-Q4257 (July 1995).
- Personnel Orientation and Training, Los Alamos National Laboratory QP-2.2.
- Standard Operating Procedure Development, Los Alamos National Laboratory QP-4.2.
- Notebook Documentation for Environmental Restoration Technical Activities, Los Alamos National Laboratory QP-5.7.
- Routine Validation of Gamma Spectroscopy Data, Los Alamos National Laboratory SOP-15.06.
- Chain of Custody for Analytical Data Packages, Los Alamos National Laboratory SOP-15.09.

10.0 ATTACHMENTS

The **user** of this SOP may locate all forms associated with this procedure at http://erinternal.lanl.gov/Quality/ user/forms.asp.

Attachment A: Laboratory Data-Validation Qualifier Flags, 1 page

Attachment B: Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data-validation Reason Codes, 3 pages

Attachment C: Data-Validation Cover Sheet, 1 page

Attachment D: Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data-Validation Checklist, 1 page

Attachment E: List of Acronyms and Abbreviations, 1 page

Attachment A: Laboratory Data-Validation Qualifier Flags

R The analyte is classified as “rejected.”

U The analyte is classified as “not detected.”

J The analyte is classified as “detected,” but the reported concentration value is expected to be more uncertain than usual.

J+ The analyte is classified as “detected,” but the reported concentration value is expected to be more uncertain than usual with a potential positive bias.

J- The analyte is classified as “detected,” but the reported concentration value is expected to be more uncertain than usual with a potential negative bias.

UJ The analyte is classified as “not detected” with an expectation that the reported result is more uncertain than usual.

Attachment B: Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data-Validation Reason Codes

Universal Reason Codes

Code	Rad	Qualifier Nondetects	Qualifier Detects	Description	Comments
1	R1	R	R	The required tracer/carrier information is missing. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
1a	R1a	R	R	The results for the affected analytes are qualified as rejected (R) because the associated tracer recovery was less than 10%.	
1b	R1b	N/A	J-	The results for the affected analytes are qualified as estimated and biased low (J-) because the associated tracer recovery was less than 30% but greater than 10%.	This code is used for detected analytes.
1c	R1c	UJ	N/A	The reporting limits for the affected analytes are qualified as estimated (UJ) because the associated tracer recovery was less than 30% but greater than 10%.	This code is used for undetected analytes.
1d	R1d	N/A	J+	The results for the affected analytes are qualified as estimated and biased high (J+) because the associated tracer recovery was greater than 105%.	Qualify only detected results.
3	R3	R	R	The required matrix spike information is missing.	The package should be returned to the SMO, or the information should be requested from the laboratory.
3a	R3a	N/A	R	The results for the affected analytes are considered rejected (R) because the associated matrix spike recovery was less than 10%.	This code is used for detected analytes.
3b	R3b	R	N/A	The reporting limits for the affected analytes are considered rejected (R) because the associated matrix spike recovery was less than 10%.	This code is used for undetected analytes.
3c	R3c	N/A	J+	The results for the affected analytes are considered estimated and biased high (J+) because the associated matrix spike recovery was above the UAL.	Qualify only detected results.

Universal Reason Codes

Code	Rad	Qualifier Nondetects	Qualifier Detects	Description	Comments
3d	R3d	N/A	J-	The results for the affected analytes are considered estimated and biased low (J-) because the associated matrix spike recovery was less than the lower acceptance limit (LAL) but greater than 10%.	This code is used for detected analytes.
3e	R3e	UJ	N/A	The reporting limits for the affected analytes are considered estimated (UJ) because the associated matrix spike recovery was less than the LAL but greater than 10%.	This code is used for (undetected) analytes.
4	R4	R (See comments)	R (See comments)	The required method-blank documentation is missing. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
4a	R4a	N/A	U	The results for the affected analytes are considered not detected (U) because the associated sample concentration was less than or equal to five times the amount in the method blank.	
5	R5	U	U	The results for the affected analytes are considered not detected (U) because the associated sample concentration was less than the MDC.	
5a	R5a	R (See comments)	R (See comments)	The MDC or TPU documentation is missing. Validation cannot proceed without this information.	The package should be returned to the SMO or the information requested from the laboratory.
6	R6	R (See comments)	R (See comments)	The LCS documentation is missing. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
6a	R6a	N/A	J+	The results for the affected analyte are considered estimated and biased high (J+) because the associated analyte in the LCS was recovered above the UAL.	Qualify only detected results.
6b	R6b	R	R	The results/reporting limits for the affected analytes should be regarded a rejected (R) because the associated LCS failed less than 10%.	
6c	R6c	N/A	J-	The results for the affected analyte are considered estimated and biased low (J-) because the associated LCS failed low but greater than 10%.	This code is used for detected analytes.
6d	R6d	UJ	N/A	The reporting limits for the affected analyte are considered estimated (UJ) because the associated LCS failed low but was greater than 10%.	This code is used for undetected analytes.

Universal Reason Codes

Code	Rad	Qualifier Nondetects	Qualifier Detects	Description	Comments
7	R7	UJ	J	The duplicate documentation is missing. Validation cannot determine the precision of the analysis without this information.	The package should be returned to SMO, or the information should be requested from the laboratory.
7a	R7a	N/A	J	The results for the affected analytes are qualified as estimated (J) because the associated duplicate sample was not prepared separately for the initial analysis.	
7b	R7b	N/A	J	The results for the affected analytes are qualified as estimated (J) because the associated duplicate sample has a DER of greater than two but less than four.	
7c	R7c	N/A	R	The results for the affected analytes are qualified as rejected (R) because the associated duplicate sample has a DER of greater than four.	
9	R9	UJ	J-	The results/reporting limits for the affected analytes are considered estimated and biased low (J-)/estimated (UJ) because the extraction holding time was exceeded.	
9a	R9a	R	R	The results for the affected analytes are considered rejected (R) because the sample extraction was double the method-published holding time.	
11	R11	N/A	U	The results for the affected analytes are considered not detected (U) because the associated sample concentration was less than three times the total propagated uncertainty.	This code should only be used after checking the detection status for results greater than the MDC.
19	R19	(See comments)	(See comments)	The validator has identified quality deficiencies in the reported data that require qualification. Please see the Data-Validation Cover Sheet for specific details.	Apply the appropriate qualifier to identify the effect of the quality deficiency on the reported data.

Attachment C: Data-Validation Cover Sheet

☐ **Rejected Data**

Section I

Request Number: _____ Validation Date: _____ Lab Code: _____

Contract Laboratory Name: _____

Validator: _____ Organization: _____

Analytical Suite (check all that apply):
☐ Volatile Organics
☐ Semivolatile Organics
☐ Organochlorine Pesticides/Polychlorinated Biphenyls
☐ High Explosives
☐ Inorganics
☐ Radiochemistry

Other (describe): _____

Section II—Completeness Check

Yes	No	n/a	(check one)	Yes	No	n/a	(check one)
☐	☐	☐	1. Chain-of-custody form(s)	☐	☐	☐	6. Raw/BSS data
☐	☐	☐	2. Case narrative	☐	☐	☐	7. Quality control forms
☐	☐	☐	3. Sample result forms	☐	☐	☐	8. Quantitation reports
☐	☐	☐	4. Sample chromatograms	☐	☐	☐	9. TICs forms
☐	☐	☐	5. Standard chromatograms	☐	☐	☐	10. TICs mass spectra

Identify any samples in the assigned Request Number that are missing:

____ ____ ____ ____ ____ ____ ____ ____

____ ____ ____ ____ ____ ____ ____ ____

Comments/problems noted (include information about requests for further information submitted to the contract laboratory and agreed-upon date of resolution and contract laboratory point-of-contact):

(Attach additional comment sheets as necessary)

Validator's signature: Date:

SOP-15.03, R1

Los Alamos National Laboratory
RRES-Remediation Program

Attachment D: RAD Checklist

Yes	No	N/A	(check one)	Assign qualifier listed below if criteria = Yes: Detected analyte	Assign qualifier listed below if criteria = Yes: Undetected analyte
☐	☐	☐	1. The MDC and/or TPU were not stated for each radionuclide in each batch for each sample.	(See note)[a]	(See note)[a]
☐	☐	☐	2. The sample result is less than the MDC.	U, R5	U, R5
☐	☐	☐	3. The sample result is less than three times the TPU calculation.	U, R11	N/A
☐	☐	☐	4. Required blank information is missing.	R, R4	R, R4
☐	☐	☐	5. The analyte detected is shown in the blank and sample result for analyte is less than or equal to the amount in blank.	U, R4a	N/A
☐	☐	☐	6. The duplicate analysis information is not present.	J, R7	UJ, R7
☐	☐	☐	7. The duplicate analysis was performed in lieu of a preparation duplicate.	J, R7a	N/A
☐	☐	☐	8. The DER for detected analytes is greater than but less than or equal to four.	J, R7b	N/A
☐	☐	☐	9. The DER for detected analytes is greater than four.	R, R7c	N/A
☐	☐	☐	10. The tracer/carrier information is not present.	R, R1	R, R1
☐	☐	☐	11. The tracer/carrier %R is greater than the UAL.	J+, R1d	N/A
☐	☐	☐	12. The tracer/carrier %R is less than 30 but greater than or equal to 10%.	J-, R1b	UJ, R1c
☐	☐	☐	13. The tracer/carrier %R is less than 10%.	R, R1a	R, R1a
☐	☐	☐	14. The LCS information is not present.	R, R6	R, R6
☐	☐	☐	15. The LCS %R is greater than 120%.	J+, R6a	N/A
☐	☐	☐	16. The LCS %R is less than 80 but greater than or equal to 10%.	J-, R6c	UJ, R6d
☐	☐	☐	17. The LCS %R is less than 10%.	R, R6b	R, R6b
☐	☐	☐	18. The matrix spike information is not present.	R, R3	R, R3
☐	☐	☐	19. The matrix spike %R is greater than 125%.	J+, R3c	N/A
☐	☐	☐	20. The matrix spike %R is less than 75% but greater than or equal to 10%.	J-, R3d	UJ, R3e
☐	☐	☐	21. The matrix spike %R is less than 10%.	R, R3a	R, R3b
☐	☐	☐	22. The sample was extracted after the appropriate holding time was exceeded.	J-, R9	UJ, R9
☐	☐	☐	23. The sample was extracted after a period double the appropriate holding time was exceeded.	R, R9a	R, R9a
☐	☐	☐	24. Other obvious data quality issues are identified.	__, R19	__, R19

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[a] If the laboratory cannot provide the missing MDC, an estimated MDC can be calculated using an amount equal to three times the TPU.

Attachment E. List of Acronyms and Abbreviations

CLP	Contract Laboratory Program (EPA)
COC	chain of custody
DER	duplicate error ratio
DLC	
EPA	U.S. Environmental Protection Agency
ER	environmental restoration
FSF	Field Support Facility
GPC	gas proportional counting
LAL	lower acceptance limit
LANL	Los Alamos National Laboratory
LCS	laboratory control sample
MDC	minimum detectable concentration
MS/MSD	matrix spike—matrix spike duplicate
n/a	not applicable
%R	percent recovery
QC	quality control
RN	request number
σ	sigma (standard deviation of a set of measurements)
SMO	Sample Management Office
SOP	standard operating procedure
SOW	statement of work
TPU	total propagated uncertainty
UAL	upper acceptable limit